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# 296 889

# AZUSA PLANT

STRUCTURAL MATERIALS DIVISION

STRESS-CORROSION CRACKING OF HIGH-STRENGTH ALLOYS

Contract DA-04-495-ORD-3069

A Report To

U.S. ARMY ORDNANCE CORPS FRANKFORD ARSENAL

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AZUSA, CALIFORNIA

This is the eighth in a series of quarterly progress reports submitted in partial fulfillment of the contract. It constitutes the second quarterly progress report for the one-year continuation of the original two year program.

This report covers the period 1 October through 31 December 1962. It was written by R. B. Setterlund, who was supervised by A. Rubin.

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#### I. OBJECTIVES

The objectives of the program extension are outlined below:

- A. Investigation of the stress-corrosion cracking characteristics of at least three new high strength alloys of interest for rocket motor case applications. These alloys will be 6Al-4V titanium, 18%-nickel maraging steel, and 20%-nickel maraging steel, in addition to limited testing of vacuum-melted 9Ni 4Co steel.
- B. Study of the environmental parameters that could affect the rate and extent of stress-corrosion cracking.
- C. Determination of the effect of material parameters (composition, strength level, welding, and microstructure) on stress-corrosion susceptibility.
- D. Continuation of the evaluation of protective coatings and other techniques for preventing stress-corrosion cracking.

#### II. SUMMARY

Data to date show that the 6A1-4V titanium alloy is immune to stress-corrosion cracking under the test conditions of this program in both the annealed and in the quenched-and-aged conditions. It has also been found that stress-corrosion cracking occurs in both 18%- and 20%-nickel maraging steels, after short exposure to specific environments. The 20%-nickel variety, if not cold-worked, is found to crack in a branching intergranular pattern when exposed to distilled water, salt water, or a high-humidity atmosphere. However, when this same alloy is cold-worked before aging, it appears to become immune to cracking in all three of these environments. The 18%-nickel maraging steel has, to date, been tested only in the cold-worked-and-aged condition. In

#### II Summary (cont.)

this condition, the alloy cracks in distilled water and in a high-humidity environment; the cracking pattern suggests possible failure along slip-planes.

#### III. WORK PROGRESS

#### A. INTRODUCTION

Since the initiation of the original test program two years ago, to investigate the stress-corrosion cracking characteristics of high-strength alloys, a number of new high-strength steels have been receiving increased attention for use in constructing rocket motor cases. The third year test program is directed to the study of three of these new alloys as well as of one titanium alloy presently being used for the same application.

The test environments, substantially the same as those evaluated in the original two-year investigation, are as follows: (1) distilled water; (2) tap water; (3) salt water; (4) sodium-dichromate-inhibited water; (5) soluble oil-inhibited water; (6) air; (7) high humidity atmosphere; (8) trichloroethylene; (9) cosmoline; and (10) solid propellant. These are considered to be environments representative of those to which rocket motor cases would normally be exposed during fabrication, processing, or storage. One additional environment will be included in the new program, that of sea coast exposure.

The test methods being used in this investigation employ bent-beam, U-bend, and center-notch specimens. Evaluation of results includes microstructural studies, using both standard metallographic and electron microscopic techniques, to attempt to associate the failure mechanism with specific microstructural characteristics of the materials.

An evaluation of protective coatings and surface treatments to prevent stress-corrosion cracking is also being conducted.

III Work Progress (cont.)

#### B. TEST PROCEDURES

#### 1. Bent-Beam Tests

The bent-beam test is the primary test method used in this program. Figure 1 shows an insulated bent-beam fixture with test samples mounted. Polycarbonate blocks  $7.000 \pm .001$  in. apart, attached to a stainless steel holder, support the test specimen and insulate it from the holder. Specimens are cut to exact length to give a maximum calculated outer fiber stress of 75% of the 0.2% offset yield strength. A four-point loading device is being used to place test specimens into the holders. By using four-point loading in this pre-stressing device, possible local plastic deformation, which may have occurred during some earlier tests with a three-point loading device, is now eliminated. Samples which have been stressed in this manner and then released show no apparent distortion.

#### 2. U-Bend Test

Figure 2 shows a U-bent test sample. This test is being used to accelerate failure times in environments where the center-notch test, described below, cannot be used.

#### 3. Center-Notch Test

Figure 3 shows the test specimen configuration used in the accelerated center-notch test. It consists of a 1-3/4- by 8-in. tensile specimen containing a central notch. This notch is produced by a two-step process. First, a 0.06- by 0.05-in. slot is Elox-machined and extended at each end by very narrow Elox-machined notches of 0.001-in. root radii. Second, an extension of these notches is produced by fatigue cycling to obtain fatigue cracks of controlled dimensions.

These center-notch specimens are tested in Baldwin creep-test machines, one of which is shown in Figure 4. Dead-weight loading is applied to a 20:1 lever arm to stress the specimen to 75% of its notched tensile strength.

III Work Progress, B (cont.)

The test solution is poured into a polyethylene cup cemented to the specimen in the notch area before the load is applied. An electric timer records the elapsed time at fracture.

#### C. PROGRAM STATUS

The titanium alloy, 6A1-4V has been under test for several weeks in two of the three conditions scheduled in Table 1. Welded plates have now been fabricated and X-rayed for joint integrity, and specimens are being prepared for testing of the remaining condition, welded joints.

The 20%-nickel maraging steel is under test in the annealed-and-aged condition as well as in the 75% cold-worked condition. The 50% cold-worked material was reannealed, by mistake, at the mill after cold-working; it was therefore necessary to prepare a new heat. Delivery of this material is scheduled for mid-February.

The 13%-nickel maraging steel is under test in the 50% cold-worked condition. Sufficient 50% cold-worked material was obtained so that part of the heat was reannealed and aged for the testing of condition I-1 (Table I). This will serve as a substitute for the annealed material on order which has not yet been received. The determination of the effect of titanium content of this alloy on its stress-corrosion cracking characteristics (another objective of this program) is being conducted with limited quantities of three additional heats obtained from another program. The chemical analysis of these heats (heats 477, 448, and 476, shown in Table 2) shows a titanium content varying from 0.40 to 1.00%.

The 9Ni-4Co vacuum cast alloy is now scheduled for delivery at the end of February. Shipment delays were caused by difficulties at the mill in producing a satisfactory heat.

III Work Program (cont.)

#### D. TEST RESULTS TO DATE

#### 1. 6Al-4V Titanium

Testing is well along on the 6Al-4V titanium alloy. The chemical analysis and mechanical properties of the material are shown in Table 3. This alloy is being tested in the three most common metallurgical conditions - annealed, quenched-and-aged, and welded. All titanium samples are given a post-heat-treat machining operation. It was found in previous work with this alloy that removal of 0.010 in. of material from each surface improves ductility, and the material is so-processed in motor case fabrication. Results of testing to date are shown in Table 4. No failures have occurred in the annealed or heat-treated conditions in any of the tests. Titanium sheets have been welded at Aerojet, the weldments X-rayed to verify their soundness, and specimens fabricated.

#### 2. 20-Nickel Maraging Steel

The chemical and mechanical properties of the 20%-nickel maraging steel are shown in Table 2. These data show that a -100°F refrigeration treatment prior to aging improved the properties of the annealed material. Environmental test results to date are shown in Table 5. The annealed-and-aged material was found to fail rapidly in both distilled water and in the 3% NaCl solution. The material also failed in the high humidity atmosphere but required a slightly longer exposure time.

Examination of the cracked specimens shows a branching crack pattern, as illustrated in Figure 5. The same figure also shows a photomicrograph of a crack which indicates cracking to be intergranular in nature.

This crack pattern, shown in Figure 6, was found to be quite slow in developing. Figure 6 drawings are based on sketches made while the crack was forming. For this particular sample, it was found that crack propagation required a longer time than did crack initiation. For this material,

#### III Work Program, D (cont.)

the center-notch tests showed roughly the same failure times in salt water and distilled water as the bent-beam tests. One bent-beam failure occurred in the dichromate solution, while two other samples in the same solution have sustained, to date, 600 hours without fracture. No failures have occurred during the center-notch testing in dichromate solution.

Two interesting results have been noted in the testing of the 20%-nickel alloy. One is the high resistance to stress-corrosion cracking of the cold-worked-and-aged material in environments where the annealed-and-aged material with lower mechanical properties cracks quickly. Another is the complete absence of cracking of either alloy, to date, in the tap water environment.

#### 3. 18-Nickel Maraging Steel

The chemical and mechanical properties of the 18%-nickel maraging steel are shown in Table 2. This heat was originally intended to be tested in the cold-worked-and-aged condition only; however, because of its relatively low titanium content we propose to test it in the annealed-and-aged condition also. Results of tests to date are shown in Table 6. Cracking in this alloy occurred most rapidly in the high humidity environment. The mode of crack propagation, however, appeared quite different from crack patterns previously encountered.

Figure 7 shows the cracking pattern on the surface of a coldworked-and-aged, 18%-nickel, bent-beam specimen after 10 days in water-saturated air, at 140°F. It depicts the multiple cracks which developed across the width of the specimen. Curiously, the direction of propagation down through the specimen thickness (shown in the bottom panel of Figure 7) is at a sharp angle from the perpendicular, forming a cross-hatch pattern; such a crack pattern is sometimes indicative of a slip-plane mode of cracking, although the information at present is insufficient to definitely establish this fact. Figure 8, a photograph, made at greater magnification, of some of these cracks indicates initiation of branching cracks developing perpendicular to the main fracture lines.

III Work Program, D (cont.)

The nature of these cracks will be further investigated to determine whether the fractures actually occur along Widmanstatten lines (indicating heat-treat or cold-working dependency) or along slip planes (indicating intercrystalline structural weakness). Residual stress measurements will be performed by X-ray diffraction methods to determine the critical stress level, and optical and electron fractography studies will be performed on the fracture surfaces to indicate the microstructural changes associated with the cracking process. A detailed examination of the cracking pattern will also be made to establish whether cracking is intergranular or transgranular in nature.

#### 4. Coatings Evaluation

The testing of various coatings designed to prevent stress-corrosion cracking in continuing (Table 7). The coatings are in addition to the three tested during the first two-year program. In the present tests, H-ll steel is being used as the base material; the test environments are % salt water, high humidity atmosphere, and sea coast exposure. H-ll steel was selected as the base material for the coating evaluation because of its characteristic short failure time, when exposed unprotected to these environments.

Presently, five coatings are under test. The 80%-aluminum, epoxy-base coating represents the principle of anodic protection by employing a metal coating that is galvanically anodic to the base material. This coating, however, appears to be unsuccessful in that short failure times are being obtained. A 70%-titanium, epoxy-base coating has been similarly unsuccessful. The zinc silicate coating under test suffered from poor adhesion and consequently afforded little protection. The 463-4-8 epoxy coating successfully protected the base metal for 500 hours of immersion in 3% salt water. (This can be compared to failure times of approximately 2 hours in earlier tests with uncoated base metal.) After this period, the coating separated from the base material, and stress-corrosion failure of the metal then followed. Epoxy coating 463-1-5, although originally developed as a primer, has been used very effectively in

III Work Program, D (cont.)

the aircraft industry, when applied as a single coating for salt water resistance. It appears to be the most promising coating in the present program, on the basis of preliminary results which show no failures in salt water after 700 hours of exposure.

#### IV. FUTURE WORK

#### A. MASTER PROGRAM

Work will be continued to fulfill the schedule shown in Table 1. Sheets of the 6A1-4V titanium alloy have been welded and specimens representing code group G-W in Table 1 will soon be in test. The annealed-and-aged 18%-nickel maraging steel (group I-1) is in the final stage of machining and specimens will be tested in January. The exposure to solid propellant and the marine atmosphere testing will be started when samples are prepared.

#### B. FURTHER EXPERIMENTS

A limited amount of additional material, consisting of three different heats of 18%-nickel maraging steel, as listed in Table 3, has been obtained from another program. This material will be used for bent-beam and center-notch specimens of each heat, in the annealed-and-aged and cold-worked-and-aged conditions, to determine the effect of titanium content of the alloy on stress-corrosion cracking. Because of the limited quantity of material available, these tests will be run in salt water and high humidity environments only.

It has been noted that to date no specimens have failed in the tap water environment. This condition could be the result of either the higher pH of the tap water (7.8), as opposed to distilled water (7.0), or to the inhibitory effect of dissolved salts in the tap water. To investigate this condition, center-notched samples of 20%-nickel maraging steel will be tested in distilled water of various pH levels.

#### IV Future Work (cont.)

#### C. METALLOGRAPHY AND ELECTRON MICROSCOPY

Photomicrographs of selected cracked specimens are being prepared and studied. In addition, the cracking process is being studied by means of the electron microscope, utilizing fracture replicas. Photographs of these replicas have been made. These results are being evaluated, while further studies are in progress. The intention is to attempt to define the mode of failure and, if possible, associate the failure process with microstructural characteristics of the materials. Evaluation of some of these results will be presented in the next quarterly report.

MASTER SCHEDULE, THIND-YEAR PROGRAM

See Coast Air Exposure (-10) (-11)	w 1 a	<b>ν</b> ια ••••	r 1	in in i	w 1 a	<b>~~</b> 1	K 1	in in i	W I Q	<b>~</b> + α	K 1	in in 1	ν·.
Solid Propellant (-09)	n ( )	MII	ĸı	MII	~ 1 1	W.L.I	٣.	ווא	~	W I I	<b>~</b> ·	<b>~</b> 1 1	ĸ.
Commodities (-08)	<b>611</b>	~	ю.	MII	ю. I т	ĸ.	٣ 1	M 1 1	M I I	MII	۲.	M I I	₩.4
Trichloro- ethylene (-07)	1 10 1	1 11/1	1 100	ıĸı	1 40 1	1101	ıĸ	I KA I	1101	IMI	• •	IMI	۱ ۳
High Humidity (-06)	WW 1	WWI	<b>~</b> ~	WW I	KN I	nn i	<b>n</b> n	WW 1	10 IO 1	in in 1	ĸĸ	NN I	КК
Soluble 011 (-05)	יי י מ	~ 1 ~	ĸΝΙ	<b>~</b> • ~	~ • ~	W 1 01	<b>K</b> ) I	<b>~</b> + α	<b>10</b>	W 1 0	ĸ	<b>M</b>   01	*^ 1 4
Sodium Dichromate Solution (-04)	יתומי	יחומ	w 1	<b>K</b> 1 0	<b>~</b> 1 0/	רומ	<b>~</b> 1	r i a	w + a	w i oi	₩.	W I OI	₩, 1
₩ ₩G (-03)	₩ I N	wia	٠, ١	<b>K</b> O L OI	~ ∙ ∾	<b>~</b> 1 01	<b>س</b> ا	W 1 0	~10	<b>n</b> • 0	٠,١	<b>*</b> \   C	٤.
(-02)	~	~ 1 1	<b>.</b> . 1	W 1 1	*^ 1 1	M) I I	<b>~</b> 1	<b>K</b> \ 1 1	W I I	M 1 1	~ 1	MII	~ 1
Distilled Water (-01)	<b>*</b> ~ · ~	~10	<b>ب</b> ۱	~ 1 0	<b>~</b> ∙ α	w 10	<b>K</b> ) 1	m i a	w i a	<b>*</b> ^ 1 (0 '	M I	<b>m</b> i a	~ •
<b>9</b> 0	G-1-B 3-1-U G-1-C	0-5-B 0-2-U 0-2-C	0-M-6	H-1-B H-1-U H-1-C	H-2-B H-2-U H-2-C	H-3-B H-3-C H-3-C	H-W-B	1-1-B 1-1-0 1-1-0	I-2-B I-2-U I-2-C	1-3-B 1-3-U 1-3-C	I-W-B I-W-U	J-I-B J-I-C J-I-C	J-2-B
Test Method	Bent Beam U-Bend Center Notch	Bent Beam U-Bend Center Notch	Bent Beam U-Bend	Bent Been U-Bend Center Notch	Bent Besm U-Bend Center Motch	Bent Besm U-Bend Center Notch	Bent Beam U-Bend	Bent Beam U-Bend Center Notch	Bent Beam U-Bend Center Notch	Bent Beam U-Bend Center Notch	Bent Beam U-Bend	Bent Beam U-Bend Center Notch	Bent Beam U-Bend
Possible Heat Treatment	As received, annealed	1650°F Wg and 900°F age	Welded, 900 <sup>O</sup> F age	Solution anneal -100°F, 850°F age	50\$ CV 850°F age	7 <b>%</b> cn 850° <b>p</b> age	Welded	of cw 0.62% Th Aged at 900°F	50% CV 0.3/0.6% Th Aged at 900°F	50% CW 0.62% Ti Aged at 900°F	0.62% Ti Welded	Aged c.25%/0.30% c	Aged 0.40/0.45% C
0.25 Yield Strength*	138,000	15,000	110,000 (anticipated)	291,000	295,000 (anticipated)	298,000	250,000 (anticipated)	283,000	500,000 (anticipated)	324,000	No data on hand	No data on hand	No data on hand
A110y	óAl-4V Titenium			20-Nickel Mareging Steel				18-NINoco Maraging Steel				9N1-4Co Vacuum-Cast Alloy	

\*\*\* Based on Aerojet tests.
\*\*\* Number refers to number of tests.
\*\*\* Lidicates material not yet received.

Table 1

TABLE 2
CHRICAL ANALYSIS AND MECHANICAL PROPRRITES OF MARAGING STEELS

Allegheny-Ludlum Heat Numbers		¥.	<u>a</u>	S	Si	Mill-Certified Analysis (Percent Composition)	Percent Co	Mo Mo	4	ಕ	77	Ħ	8	<u>m</u>
0000		0.09 0.002 0.002	0.000 0.000 0.000 0.000	0.005 0.005 0.005	8000 1800 1800 1800 1800 1800 1800 1800	ដូច្ចិត្ត សូច្ចិត្ត សូច្ចិត្ត	. 6. 6. 4. 6. 6. 4.	4 4 4	6 6 6 6 6 6 6 6 6 6 6 6 6 6 6 6 6 6 6	3.39	0.002	400c	₹80°. 20°.	0.003
; ·	0.020	3.30%	oc.		0.00 0.014 Wechanica	0.00 15.50 5.05 +:5 Wechanical Properties (Aerolet Tests)	3.05 Aerolet	1.3c	0.078			100		1 1
불씨	Percent Cold Reduction		Aging Treatment	Table 1	No.	0.2% Offset Y.S. (psi) (Transverse)	S. (pst)	U.T.S. (psi) (Transverse)		Percent Elongation (in 2 in.)		Notobed Tensile Strength (psi)	-	Haraa Rockwell 37
	34 N N N N N N N N N N N N N N N N N N N	1.001. 3.26 3.86	None -120°F + 350°F + ar None 350°F thr None	nr H-1 H-2 H-3		128,500 291,300 185,100** 319,200** 205,700 298,300	* *	170, 700 302, 200 197, 903** 322, 700** 220, 900 308, 300	* *	5.5 *** 1.5 ° ° ° ° ° ° ° ° ° ° ° ° ° ° ° ° ° ° °		33,200 - - - 31,500		** ***********
	\$ 0.00 20.00 20.00	Ř Ř	None 300°F 3 hr None 300°F 3 hr	I-1 I-3	,	132,360 283,000 167,700 323,800		153,300 294,300 189,000 328,400		% <b>%</b> 17.88				S WANT
	55 55	3	None			109,300 278,000		196,900 230,730		0.5 10.				10 m 10 m 10 m
	<b>2000</b>	8 8	None 900°F 3 hr None 900°F 3 hr			105,300 255,400 175,500 331,000		150,300 265,900 199,300 332,500		01 0.4.1 1.1.0		1 1 1 1		50 85 85 15. 5. 63 85 15.
	0000	8 8	None 900'F 3 hr None 900'F 3 hr	1111		128, 500 323, 500 192, 200 354, 400		174,700 530,000 217,000 354,300		regard review				35 to 35

\*\*\* Tensile tests of fatigue-cracked specimens shown in Figure 5.
\*\*\* Mill report.
\*\*\*\* Re-annealed, cold-worked material.

Table 2

TABLE 3

CHEMICAL ANALYSIS AND MECHANICAL PROPERTIES

OF 6A1-4V TITANIUM

			Che	mical Ana	alysis (9	Composi	$tion)^*$		
	C	Al	<u>v</u>	02	_N <sub>2</sub>	H <sub>2</sub>	<u>Ti</u>	<u>Fe</u>	Other
Aerojet Analysis	0.03	6.1	4.1	0.083	0.014	80 ppm	Bal	0.16	0.18

	Mechanica	l Properti	es (Transver	·se)
	Yield Strength (0.2% offset) (psi)	Ultimate Strength (psi)	Elongation (%)	$\frac{\texttt{Rc})}{\texttt{Rc}}$
Annealed				
Mill report	131,900	141,400	12	33.5
Aerojet test	138,000	143,800	14	34
Notched tensile strength **		128,500	-	-
1675°F 1 hr, W.Q. Aged 900°F 8 hr				
Aerojet test	162,700	176,800	7	38.5
Notched tensile strength		132,000	-	-

<sup>\*</sup>Titanium Metals Corporation HT 4141.

<sup>\*\*</sup> Using as-fatigue-cracked sample of Figure 3.

TABLE 4

STRESS CORROSION OF 6A1-4V TITANIUM
IN VARIOUS ENVIRONMENTS

		aled ceived	.)	Conditi 1675 F 1 h 900 F	r. W.G	
		Fail	ure Times		Failur	e Times
Environment	Failed/Tested	Mean (hr)	Range (hr)	Failed/Tested	Mean (hr)	Range (hr)
Bent Beam Tests						
Distilled water Tap water 3% NaCl sol. 0.25% Sodium dichromate Soluble oil sol. Cosmoline High-humidity atmosphere Air Solid propellant Sea-coast exposure	0/3** 0/3 0/3 0/3 0/2 0/3 0/3 0/3 0/3****	-	NF780***  NF780 -	0/3 0/3 0/3 0/3 0/3 0/3 0/3 0/0 0/0	-	NF780 NF780
U-Bend Tests						
High-humidity atmosphere Trichloroethylene Sea-coast exposure	0/0 0/0 0/0	- - -	- - -	0/3 0/0 0/0	- - -	NF310 - -
Center-Notch Tests						
Distilled water 3% NaCl sol. 0.25% Sodium dichromate Soluble oil sol. (4%)	0/2 0/2 0/2 0/1	- - -	NF100 NF100	0/2 0/2 0/2 0/1	- - -	NF100 NF100

Refers to code letter in Master Schedule, Table 1.

<sup>\*\*</sup> Indicates no failures of three samples exposed.

<sup>\*\*\*</sup> Indicates no failures in 780 hours exposure.

<sup>\*\*\*\*</sup> Indicates testing not started.

TABLE 5

STRESS CORROSION OF 20%-NICKEL
MARAGING STEEL IN VARIOUS ENVIRONMENTS

	Conditation Solution Conditation Conditati	on Anne	al	Conditi 75% Col 850 F	d Work	
	<del></del>	Fail Mean	ure Times Range		Failur Mean	e Times Range
Environment	Failed/Tested		(hr)	Failed/Tested	(hr)	(hr)
Bent Beam Tests						
Distilled water	3/3**	11	10.2-18	0/3	-	NF600
Tap water	0/3	-	NF600***	0/3	-	
3% NaCl sol.	3/3	7.3		0/3	-	
0.25% Sodium dichromate Soluble oil sol.	1/3 0/3	1	1-nf600 nf600	0/3 0/3	_	
Cosmoline	0/3	_	NF600	0/3	-	
High-humidity atmosphere	3/3	100		0/3	-	$\checkmark$
Air	0/3	-		0/3	-	nf600
Solid propellant	0/0***	_	-	0/0	-	-
Sea-coast exposure	0/0	-	-	0/0	-	-
U-Bend Tests						
High-humidity atmosphere	0/0	-	-	0/0	-	-
Trichlorethylene	0/0	-	-	0/0	-	-
Sea-coast exposure	0/0	-	-	0/0	-	-
Center-Notch Tests						
Distilled water	3/3		4.3-6.6	0/0	-	-
3% NaCl sol.	2/2	7.2	6.6 <b>-</b> 7.8	0/,0	-	-
0.25% Sodium dichromate	0/2	-	<b>NF</b> 60	0/0	-	-
Soluble oil sol. $(4\%)$	0/1	-	nf60	0/0	-	-
Air	0/0	-	-	0/0	-	-

<sup>\*</sup>Refers to code letter of Table 1, Master Schedule.

<sup>\*\*</sup> Indicates three failures of three samples tested.

<sup>\*\*\*\*</sup> Indicates no failures in 600 hours of testing.

<sup>\*\*\*\*</sup> Indicates testing not started.

TABLE 6

STRESS CORROSION OF 18%-NICKEL
MARAGING STEEL IN VARIOUS ENVIRONMENTS

Environment Bent Beam Tests	Condition 50% C.W., 1500°F 1 hr, Ag <u>I</u> Failed/Tested	Annea ged 90 Failur Mean	led OF 3 hr e Times Range (hr)		Mean	W.
Distilled water Tap water 3% NaCl 0.25% Sodium dichromate Soluble oil sol. High-humidity atmosphere Cosmoline Solid propellant Air Sea-coast exposure	0/0** 0/0 0/0 0/0 0/0 0/0 0/0 0/0 0/0 0/	-	- - - - - -	1/2*** 0/2 0/2 0/2 0/2 0/2 2/2 0/2 0/0 0/0	440 - - - 260 - -	440-NF445**** NF445 NF445 NF445 NF445 245-290 NF445
U-Bend Tests  High-humidity Trichloroethylene Sea-coast exposure	0/0 0/0 0/0	- - -	- - -	o/o o/o o/o	- - -	- - -
Center-Notch Tests  Distilled water 3% NaCl sol. 0.25% Sodium dichromate Soluble oil (4% sdn) Air	0/0 0/0 0/0 0/0 0/0		- - - -	o/o o/o o/o o/o o/o	- - -	- - - -

<sup>\*</sup>Refers to code letter of Table 1, Master Schedule.

<sup>\*\*</sup> Indicates tests not started.

<sup>\*\*\*</sup> Indicates one failure of two samples tested.

<sup>\*\*\*\*</sup> Indicates 445 hours without failure.

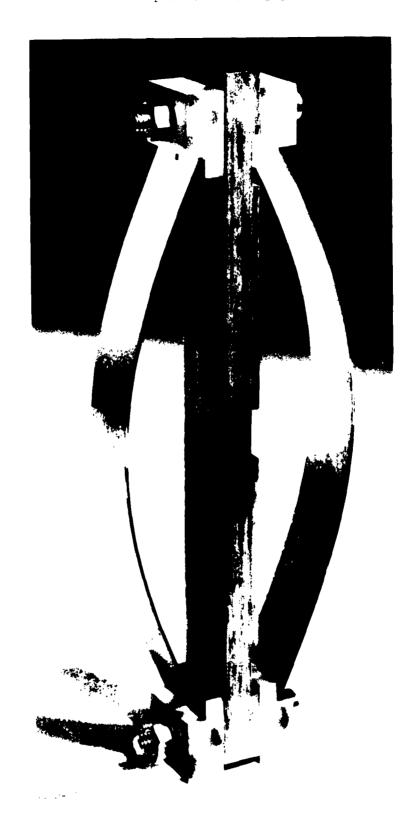
TABLE 7

EVALUATION OF PROTECTIVE COATINGS ON H-11 STEEL (FOR PREVENTING STRESS-CORROSION CRACKING)

	140°F High Humi			3% NaCl		
	Failed/Tested	Failu Mean (hr)	re Times Range (hr)	Failed/Tested	Failu Mean (hr)	Range (hr)
H-11 Steel (uncoated)	2/2 <del>*</del>	64	48 <b>-</b> 70	2/2	1.7	0.8-2.5
Epoxy 463-4-8	1/3	289	289 <b>-</b> NF570**	3/3	550	525-578
Epoxy 463-1-5	1/3	400	400 <b>-NF</b> 570	0/3	-	NF700
Zinc silicate	1/2	170	170-NF440	2/2	1.2	0.8-1.6
80% Aluminum-epoxy	2/2	30	16-45	2/2	100	100
70% Titanium-epoxy	1/2	150	150 <b>-NF</b> 240	2/2	150	140-160

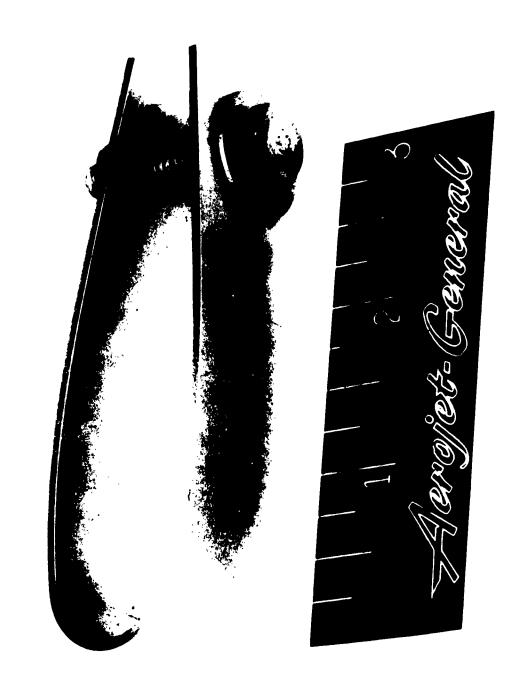
<sup>\*</sup>Indicates two failures out of two samples exposed.

<sup>\*\*</sup> Indicates no failure in 570 hours.

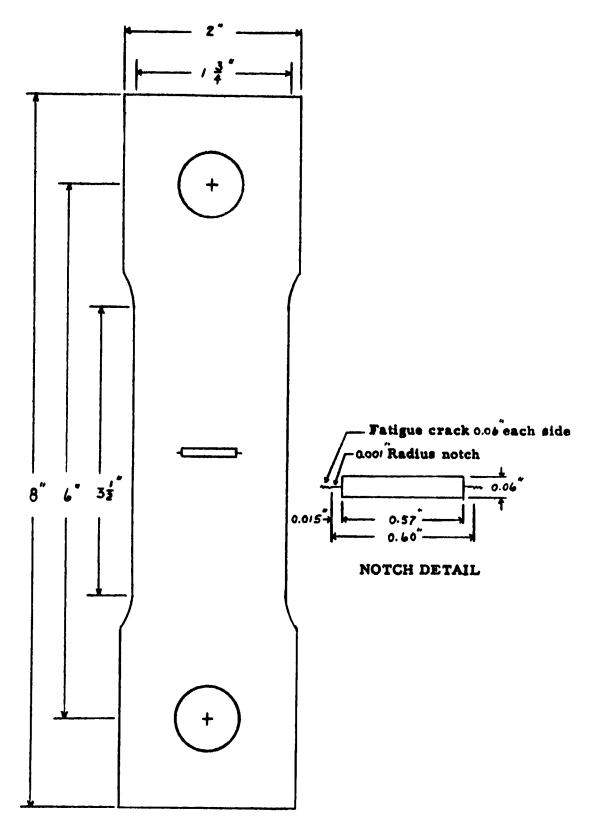


Insulated Phelps Bent-Beam Specimens

Firar 1

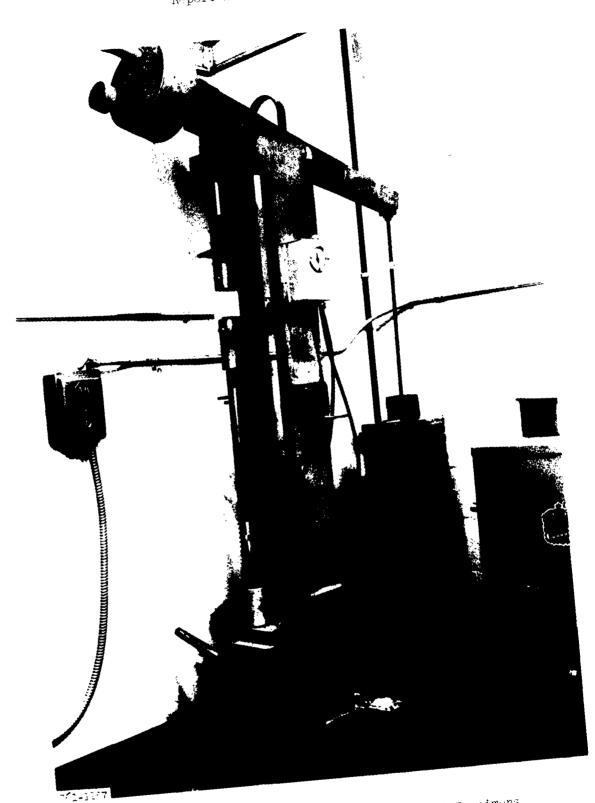


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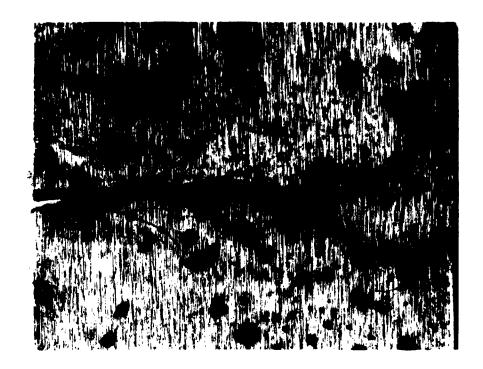


ELOX-NOTCHED SPECIMEN FOR CRACK PROPAGATION STUDY

Figure 3



Stress-Corresion Test Setup for Center-Netched Specimens



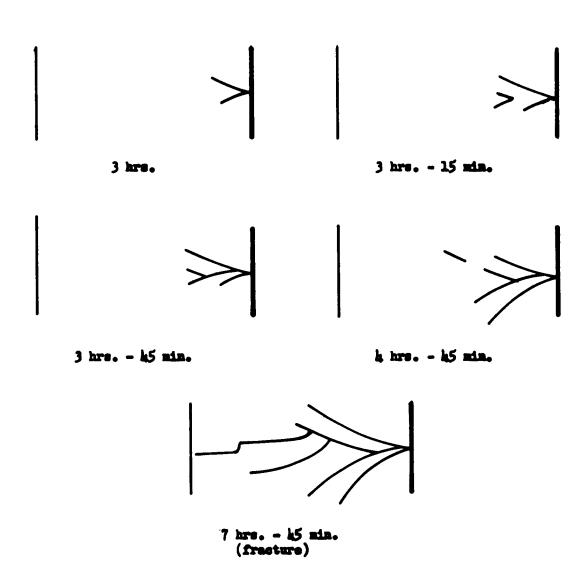
Crack Pattern on Surface of Beam Sample after 10 hours in Aerated Distilled Water. (5%)



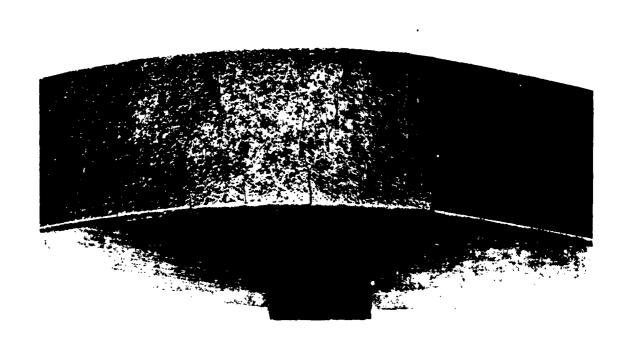
Vertical View of Cracking in Above Sample Showing Intergranular Cracks. Etchant is Diluted Marbles Reagent (1000X)

Stress-Corrosion Crack Pattern on 20%-Nickel Maraging Steel

Figure 5



Crack Propagation Study on 20%-Nickel Maraging Steel in Salt Water

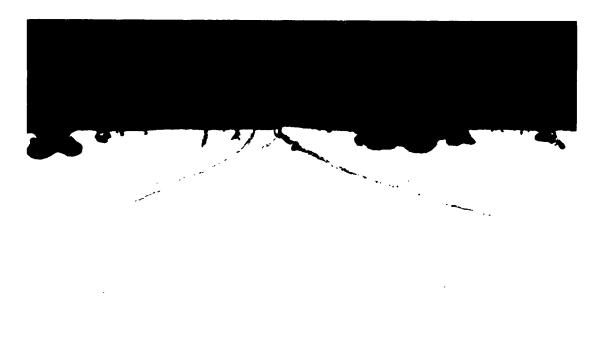


Surface of Cold-Worked-and-Aged 18%-Nickel Maraging Steel After 10-Days at 140°F in High Humidity Stress Corrosion Test. Surface has been Wire-Brushed. (Approx. 2X)



Cross-Section of Above Sample Showing Possible Cracking Along Slip Planes. (10X)

Stress-Corrosion Crack Pattern on 20%-Nickel Maraging Steel



View of Surface in Lightly Cracked Area Showing Pitting Attack (100X)



General Structure in Interior of Highly Cracked Area. Etchant - 104 Ammonium Persulphate-electrolytic. (500X)

Phot mirr graphs of Stress-Corresion Creeking in 18"-Nickel Maraging Steed



## **AEROJET-GENERAL CORPORATION**